

USEPA Region II
SW846 Method 8080A/8000A

SOP HW-23, Rev. 0

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PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: _____ LAB: _____

SITE: _____

1.1 Has all the data been submitted in CLP deliverable format?

1.2 Have any missing deliverables been received and added to the data package?

2.1 Is a laboratory narrative or cover letter present?

2.2 Are the case number and/or SDG number contained in the narrative or cover letter?

3.1 Does this data package contain:

Water data?

Waste data?

Soil/solid data?

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YES NO N/A

[illegible]

1.0 Traffic Reports and Laboratory Narrative

ACTION: If samples were not iced or if the ice was melted upon arrival at the laboratory and the temperature of the cooler was elevated (> 10E C), flag all positive results "J" and all non-detects "UJ".

2.1 Have any organochlorine pesticide/PCB technical holding times, determined from date of collection to date of extraction, been exceeded? ☐ ☐

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YES	NO	N/A
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YES NO N/A

limits been calculated properly using the procedure outlined in section 8.10, pages 8000A-13 & 14? [

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YES NO N/A

ACTION: If evidence suggests that the surrogate control limits were calculated improperly, contact the laboratory for explanation. Make note of the problem in the data assessment and qualify data based on 60-150% recovery in section 3.4 below.

ACTION: Circle all outliers in red.

3.5 Were surrogate retention times (RT) within the windows established during the initial 5-point analysis? []

3.6 Are there any transcription/calculation errors between raw data and Form II? _____ [] _____

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YES	NO	N/A
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corrections and document the effect in data assessments.

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YES NO N/A

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4.3 Do standard deviation (s) and average recovery⁻(x in Fg/l) for each analyte meet the corresponding QC acceptance criteria listed in Table 3, page 8080A-17?

- 1) Locate and correct the source of the problem; reextract and reanalyze 4 new QC check samples containing all analytes of interest.
- 2) Reextract and reanalyze 4 QC check samples containing only those analytes which failed the initial test.

5.0 Matrix Spikes (Form III)

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YES NO N/A

replicate samples in place of the matrix spike (see page 8000A-11, section 8.7).

5.2 Have MS/MD or MS/MSD results been summarized on modified CLP Form III?

ACTION: If any data are missing take action as specified in section 3.2 above.

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YES NO N/A

a. Water [] ____

b. Waste [] ____

c. Soil/Solid []

Criteria used

<u>Waters</u>		<u>Wastes</u>		<u>Soils/Solids</u>	
out of	*	out of	*	out of	*

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YES	NO	N/A
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YES NO N/A

5.6 Was the matrix spike prepared at the proper concentration?

[]

For aqueous organic extractibles, the spike concentration should be:

- 1) For regulatory compliance monitoring - the regulatory concentration limit or 1 to 5 times the expected background concentration, whichever is higher;
- 2) For all other aqueous samples - the larger of either 1 to 5 x times the expected background concentration, or the same as the QC check sample concentration (see section 4 above);
- 3) For soil/solid and waste samples - the recommended concentration is 20 times the estimated quantitation limit (EOL).

ACTION: No action is taken based on MS or replicate data alone. However, using informed professional judgement, the data reviewer may use the matrix spike or laboratory replicate results in conjunction with other QC criteria and determine the need for some qualification of the data. In some instances it may be determined that only the replicate or spiked samples are affected. Alternatively, the data may suggest that the laboratory is having a systematic problem with one or more analytes, thereby affecting all associated samples.

6.0 Blanks (Form IV)

6.1 Was reagent blank data reported on CLP Method Blank Summary form(s) (Form IV)?

[]

6.2 Frequency of Analysis: For the analysis of organochlorine pesticide/PCB compounds, has a reagent blank been analyzed for every 20 samples of similar matrix or concentration or each

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YES	NO	N/A
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extraction batch, whichever is more frequent?

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                                     YES    NO    N/A
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YES NO N/A

materials: Supelcoport (100/120 mesh), coated with 1.5% SP-2250/1.95% SP-2401, packed in a 1.8 m x 4 m ID glass column or equivalent?

Was column 2 constructed using the recommended Supelcoport (100/120 mesh), coated with 3% OV-1 in a 1.8 m x 4 mm ID glass column or equivalent

? [] _____

[illegible]

pesticides? _____

PCBs? _____

column 1: _____

column 2: _____

9.0 Calibration and GC Performance

a.	DDT/endrin breakdown check	<input type="checkbox"/>	___	___
b.	Aroclor 1016/1260	<input type="checkbox"/>	___	___
b.	Aroclors 1221, 1232, 1242, 1248, 1254	<input type="checkbox"/>	___	___
c.	toxaphene	<input type="checkbox"/>	___	___
d.	technical chlordane	<input type="checkbox"/>	___	___
e.	5 pt. initial calibration standards	<input type="checkbox"/>	___	___
f.	calibration verification standards	<input type="checkbox"/>		

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YES NO N/A

h. reagent blanks

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YES NO N/A

- 9.4 Are data summary forms (containing calibration factors or response factors) for the initial 5 pt. calibration and daily calibration verification standards present and complete for each column and each analytical sequence? _____

ACTION: If any data are missing or it cannot be determined how the laboratory calculated calibration factors or response factors, contact the lab for explanation/resubmittals. Make necessary corrections and note any problems in the data assessment.

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document the effect in data assessments.

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YES NO N/A

9.6 Are standard retention time (RT) windows for each analyte of interest presented on modified CLP summary forms? [

ACTION: If any data are missing, or it cannot be determined how RT windows were calculated, call the lab for explanation/resubmittals. Note any problems in the data assessment.

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YES NO N/A

A 72 hr. sequence is not required with this method (page 8080A-7, section 7.4.2); however, the method states that best results are obtained using retention times which span the entire sequence. I.E., using the mid level from the 5 pt., one of the mid-concentration standards analyzed during mid-sequence and one analyzed at the end.

[]

ACTION: Note potential problems, if any, in the data assessment.

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YES	NO	N/A
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YES NO N/A

[illegible]

9.12 Has the percent difference (%D) exceeded $\pm 15\%$ for any organochlorine pesticide/PCB analyte in any calibration verification standard or mid-concentration standard? _____ [] _____

9.13 Has a new 5 pt. calibration curve been generated for those analytes which failed in the calibration verification standard (page 8000A-3, section 7.4.2.3), and all samples which followed the out-of-control standard (page 8000A-6, section 7.6.8) reinjected?

ACTION: If the %D for any analyte exceeded the $\pm 15\%$ criterion and the instrument was not recalibrated for those analytes, qualify positive results for all associated samples (those which followed the out-of-control standard) "J" and sample quantitation limits "UJ". If the %D was $> 90\%$ for any analyte, qualify non-detects "R", unusable.

9.14 Have daily retention time windows been properly calculated for each analyte of interest (page 8000A-6, section 7.6.9.), using RTs from the associated mid concentration standard and standard deviation from the initial calibration)?

ACTION: If no, take action specified in section 3.2 above or recalculate RT windows using the procedure outlined in method 8000A-5, section 7.5.2.2.

9.15 Do all standard retention times for each mid concentration standard fall within the windows established during the initial calibration sequence? [] [] []

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YES NO N/A

10.0 Analytical Sequence Check (Form VIII-PEST)

10.1 Have all samples been listed on CLP Form VIII or equivalent, and are separate forms present for each column? []

ACTION: If no, take action specified in 3.2 above.

10.2 Was the proper analytical sequence followed for each initial calibration and subsequent analyses (see pages 8080A-6 & 7, section 7.4)? []

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify it accordingly. Generally, the effect is negligible unless the sequence was grossly altered or the calibration was also out of limits.

11.0 Cleanup Efficiency Verification (Form IX)

11.1 Is Form IX - Pest-1 present and complete for each lot of Florisil/Cartridges used? (Florisil Cleanup, Method 3620A, is required for all organochlorine pesticide/PCB extracts.) []

ACTION: If no, take action specified in 3.2 above. If data suggests that florasil cleanup was not performed, make note in the reviewer narrative.

NOTE: Method 3620A uses florisisl, while the SOW/CLP allows for florisisl cartridges. Method 3620A does not list which pesticides and surrogate(s)

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                                           YES   NO   N/A
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If only PCBs are to be measured in a sample, the sulfuric acid/permanganate cleanup method (Method 3665), followed by Silica Cleanup (Method 3630) or Florisil Cleanup (Method 3620) is recommended.

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YES NO N/A

12.3 Are retention times (RT) of sample compounds within the established RT windows for both analyses?

ACTION: Qualify as unusable (R) all positive results which were not confirmed by second GC column analysis. Also qualify "R", unusable, all positive results not within RT windows unless associated standard compounds are similarly biased. The reviewer should use professional judgement to assign an appropriate quantitation limit.

12.4 Check chromatograms for false negatives, especially if RT windows on each column were established differently (see section 9.7 above). Also check for false negatives among the multiple peak compounds toxaphene, chlordane and PCBs. Were there any false negatives?

[]

ACTION: Use professional judgement to decide if the compound should be reported. If there is reason to believe that peaks outside retention RT windows should be reported, make corrections to data summary forms (Form I) and note in data assessment.

12.5 Was GC/MS confirmation provided when sample concentration was sufficient (> 10 ug/ml) in the final extract?

[]

ACTION: Indicate with red pencil which Form I results were confirmed by GC/MS and also note in data assessment.

12.6 Is the percent difference (%D) calculated for the positive sample results on the two GC columns <25.0%?

[]

NOTE: The method 8080A requires quantitation from one column. The second column is to confirm the

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YES NO N/A

presence of an analyte. Calibration for the Confirmation column is a one point calibration. It is the reviewer's responsibility to verify from the project plan what the lab was required to report. If the lab was required to report concentrations from both columns, continue with validation for % Difference. If required, but not reported, either contact the lab for results or calculate the concentrations from the calibration. If not required, skip this section. Document actions in Data Assessment.

ACTION: If the reviewer finds neither column shows interference for the positive hits, the data should be qualified as follows:

<u>% Difference</u>	<u>Qualifier</u>
0-25%	none
25-70%	"J"
70-100%	"NJ"
>100%	"R"
100-200% (Interference detected)*	"NJ"
>50% (Pesticide vale is <CROL)**	"U"

* When the reported %D is 100-200% but interference is detected in either column, qualifiy the data with "NJ".

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**      When the reported pesticide value is
lower than the CRQL, and the %D is
>50%, raise the value to the CRQL and
qualifiy with "U" (non-detect).

```

13.0 Compound Quantitation and Reported Detection Limits

13.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Were any errors found?

NOTE: Single-peak pesticide results can be checked for rough agreement between quantitative results obtained on the two GC columns. The

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YES NO N/A

14.0 Chromatogram Quality

14.1 Were baselines stable?

14.2 Were any electropositive displacement
(negative peaks) or unusual peaks seen? _____ [] _____

ACTION: Note all system performance problems in the data assessment.

15.0 Field Duplicates

15.1 Were any field duplicates submitted for organochlorine pesticide/PCB analysis? 1

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, the identity of the field duplicates is questionable. An attempt should be made to determine the proper identification of field duplicates.